



DATA VALIDATION REPORT

Gold King Mine Release Incident

SAMPLE DELIVERY GROUP: 680-117013-2

Prepared by

MEC^X
12269 East Vassar Drive
Aurora, CO 80014



I. INTRODUCTION

Task Order Title: Gold King Mine Release Incident
Project No.: 20408.012.001.0274.00
20408.012.001.0267.00
Sample Delivery Group: 680-117013-2
EPA Project Manager: Steve Way
Weston Project Manager: Dave Robinson
TDD No.: 0001/1508-04
Matrix: Sediment
QC Level: Stage 2A
No. of Samples: 1
No. of Reanalyses/Dilutions: 0
Laboratory: TestAmerica – West Sacramento

Table 1. Sample Identification

| Location ID | Lab Sample Name | Matrix Type | Collection Date | Method |
|------------------|-----------------|-------------|-----------------|--------|
| CC06_092115_1300 | 680-117013-1 | Sediment | 9/21/15 1:00 PM | 8290A |

II. Sample Management

Anomalies regarding sample management are noted below. The sample was received below the temperature limits of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, at 0.6°C at TestAmerica – Denver, and 0.3°C at TestAmerica – West Sacramento; however, as the sample was not noted to be frozen or damaged, no qualification was necessary. The sample was received intact, on ice, and properly preserved. The chains-of-custody (COCs) were appropriately signed and dated by field and laboratory personnel. The presence or absence of custody seals on the cooler was not specifically noted.

The following issues were noted:

- ☐ Numerous corrections made to the original COC were made by overwriting the original entry. Most corrections were initialed; however, most corrections were not dated.
- ☐ The location ID was listed incorrectly on the COCs as CC06_09212015_1300.
- ☐ The COCs did not list CLP sample IDs, and none were provided. The laboratory logged the sample per the location ID on the COCs.
- ☐ The presence or absence of sample tags was not noted in the case narrative, and sample tags were not listed on the COCs.

**Data Qualifier Reference Table**

| Qualifier | Organics | Inorganics |
|-----------|--|--|
| U | The analyte was analyzed for, but was not detected above the reported sample quantitation limit. The associated value is the quantitation limit or the estimated detection limit for dioxins or PCB congeners. | The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit. The associated value is the sample detection limit or the quantitation limit for perchlorate only. |
| UB | The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination. | The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination. |
| J | The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample. | The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample. |
| J+ | Not applicable | The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential positive bias. |
| J- | Not applicable | The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential negative bias. |



| Qualifier | Organics | Inorganics |
|-----------|---|---|
| UJ | The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample. | The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise. |
| UJB | The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample. | The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample. |
| N | The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification." | Not applicable. |
| NJ | The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration. | Not applicable. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified. | The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified. |



Qualification Code Reference Table

| Qualifier | Organics | Inorganics |
|-----------|--|---|
| H | Holding times were exceeded. | Holding times were exceeded. |
| S | Surrogate recovery was outside QC limits. | The sequence or number of standards used for the calibration was incorrect |
| C | Calibration %RSD or %D was noncompliant. | Correlation coefficient is <0.995 or calibration was noncompliant. |
| R | Calibration RRF was <0.05. | %R for calibration is not within control limits. |
| B | Presumed contamination as indicated by the preparation (method) blank results. | Presumed contamination as indicated by the preparation (method) or calibration blank results. |
| L | Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits. | Laboratory Control Sample %R was not within control limits. |
| L1 | LCS/LCSD RPD was outside control limits. | LCS/LCSD RPD was outside control limits. |
| Q | MS/MSD recovery was poor. | MS recovery was poor. |
| Q1 | MS/MSD RPD was outside control limits. | MS/MSD RPD was outside control limits. |
| E | Not applicable. | Duplicates showed poor agreement. |
| I | Internal standard performance was unsatisfactory. | ICP ICS results were unsatisfactory. |
| A | Not applicable. | ICP Serial Dilution %D were not within control limits. |
| M | Tuning (BFB or DFTPP) was noncompliant. | ICPMS tune was not compliant. |
| T | Presumed contamination as indicated by the trip blank results. | Not applicable. |
| + | False positive – reported compound was not present. | Not applicable. |
| - | False negative – compound was present but not reported. | Not applicable. |
| F | Presumed contamination as indicated by the FB or ER results. | Presumed contamination as indicated by the FB or ER results. |
| F1 | Field duplicate results were outside the control limit. | Field duplicate results were outside the control limit. |
| \$ | Reported result or other information was incorrect. | Reported result or other information was incorrect. |



| Qualifier | Organics | Inorganics |
|-----------|--|--|
| ? | TIC identity or reported retention time has been changed. | Not applicable. |
| D | The analysis with this flag should not be used because another more technically sound analysis is available. | The analysis with this flag should not be used because another more technically sound analysis is available. |
| P | Instrument performance for pesticides was poor. | Post Digestion Spike recovery was not within control limits. |
| *II, *III | Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found. | Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found. |



III. Method Analyses

A. EPA Method 8290A— Dioxin/Furans

Reviewed By: L. Calvin

Date Reviewed: October 14, 2015

The sample listed in Table 1 for this analysis was validated based on the guidelines outlined in the *Sampling and Analysis Plan/Quality Assurance Project Plan for Gold King Mine Release, Silverton, San Juan County, Colorado* (2015), *United States Environmental Protection Agency Contract Laboratory Program Statement of Work for Organic Superfund Methods, EPA Method SW-846 8290A, Contract Laboratory Program Statement of Work for Organic Superfund Methods*, and the *National Functional Guidelines for Chlorinated Dioxin and Furan Data Review* (2011).

- ☐ Holding Times: The sediment sample was extracted within 30 days of collection and analyzed within 45 days of extraction.
- ☐ Analytical Method Blanks: The method blank had detects below the reporting limits for 1,2,3,4,6,7,8-HpCDD (0.144 pg/g), 2,3,4,6,7,8-HxCDF (0.120 pg/g), OCDD (0.630 pg/g), OCDF (0.962 pg/g), and totals HpCDD and HxCDF. Sample results below the reporting limits for the isomer method blank contaminants were qualified as nondetected (UB) at the level of contamination. The sample results for totals HpCDD and HxCDF were qualified as estimated (J), as the portion of the totals due to method blank contamination could not be determined.
- ☐ Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD): Recoveries were within the laboratory-established control limits, and RPDs were within the control limit of $\leq 20\%$.
- ☐ Matrix Spike/Matrix Spike Duplicate (MS/MSD): MS/MSD analyses were not performed on the sample in this SDG.
- ☐ Field QC Samples: Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site samples. Following are findings associated with field QC samples:
 - Field Blanks and Equipment Rinsates: This SDG had no identified field blank or equipment rinsate samples.
 - Field Duplicates: This SDG had no identified field duplicate samples.



- ☐ Internal Standards Performance: The labeled standard recoveries were within the control limits of 40-135%.
- ☐ Compound Identification: A detect not meeting the method ion abundance ratio criteria was flagged by the laboratory as an estimated possible concentration (EMPC). Isomers flagged as EMPCs were qualified as estimated nondetects (UJ) at the level of the EMPC. Totals flagged as containing one or more EMPC peaks were qualified as estimated (J).

Validated Sample Result Forms: 680-117013-2

Analysis Method 8290A

Sample Name CC06_092115_1300

Matrix Type: Solid

Lab Sample Name: 680-117013-1

Sample Date: 9/21/2015 1:00:00 PM

| Analyte | Total/Dissolved | CAS No | Result Value | Reporting Limit | MDL | Result Units | Lab Qualifier | Validation Qualifier | Validation Notes |
|---------------------|-----------------|------------|--------------|-----------------|------|--------------|---------------|----------------------|------------------|
| 1,2,3,4,6,7,8-HpCDD | T | 35822-46-9 | 28 | 28 | 0.27 | pg/g | J B | UB | B |
| 1,2,3,4,6,7,8-HpCDF | T | 67562-39-4 | 2.2 | 28 | 0.57 | pg/g | J | J | |
| 1,2,3,4,7,8,9-HpCDF | T | 55673-89-7 | 2.7 | 28 | 0.7 | pg/g | J q | UJ | *III |
| 1,2,3,4,7,8-HxCDD | T | 39227-28-6 | 1.2 | 28 | 0.21 | pg/g | J q | UJ | *III |
| 1,2,3,4,7,8-HxCDF | T | 70648-26-9 | 1.5 | 28 | 0.3 | pg/g | J q | UJ | *III |
| 1,2,3,6,7,8-HxCDD | T | 57653-85-7 | 1.7 | 28 | 0.2 | pg/g | J | J | |
| 1,2,3,6,7,8-HxCDF | T | 57117-44-9 | 1.9 | 28 | 0.27 | pg/g | J | J | |
| 1,2,3,7,8,9-HxCDD | T | 19408-74-3 | 1.9 | 28 | 0.18 | pg/g | J | J | |
| 1,2,3,7,8,9-HxCDF | T | 72918-21-9 | 2.5 | 28 | 0.31 | pg/g | J | J | |
| 1,2,3,7,8-PeCDD | T | 40321-76-4 | 0.36 | 28 | 0.36 | pg/g | U | U | |
| 1,2,3,7,8-PeCDF | T | 57117-41-6 | 0.9 | 28 | 0.24 | pg/g | J q | UJ | *III |
| 2,3,4,6,7,8-HxCDF | T | 60851-34-5 | 28 | 28 | 0.29 | pg/g | J B | UB | B |
| 2,3,4,7,8-PeCDF | T | 57117-31-4 | 0.87 | 28 | 0.25 | pg/g | J q | UJ | *III |
| 2,3,7,8-TCDD | T | 1746-01-6 | 0.44 | 5.6 | 0.24 | pg/g | J q | UJ | *III |
| 2,3,7,8-TCDF | T | 51207-31-9 | 0.21 | 5.6 | 0.21 | pg/g | U | U | |
| OCDD | T | 3268-87-9 | 56 | 56 | 0.29 | pg/g | J B | UB | B |
| OCDF | T | 39001-02-0 | 56 | 56 | 0.55 | pg/g | J B | UB | B |
| Total HpCDD | T | 37871-00-4 | 3.2 | 28 | 0.27 | pg/g | J B | J | B |
| Total HpCDF | T | 38998-75-3 | 4.9 | 28 | 0.64 | pg/g | J q | J | *III |
| Total HxCDD | T | 34465-46-8 | 4.7 | 28 | 0.2 | pg/g | J q | J | *III |
| Total HxCDF | T | 55684-94-1 | 8.2 | 28 | 0.29 | pg/g | J q B | J | B, *III |
| Total PeCDD | T | 36088-22-9 | 0.36 | 28 | 0.36 | pg/g | U | U | |
| Total PeCDF | T | 30402-15-4 | 1.8 | 28 | 0.25 | pg/g | J q | J | *III |
| Total TCDD | T | 41903-57-5 | 0.44 | 5.6 | 0.24 | pg/g | J q | J | *III |
| Total TCDF | T | 30402-14-3 | 0.21 | 5.6 | 0.21 | pg/g | U | U | |